Identifier:	Revision:	Effective Date:
ER-SOP-15.06	**0**	**7/20/00**

ER Document Catalog Number: **ER2000-0236**

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environmental restoration project

A Department of Energy
Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

Routine Validation of Gamma Spectroscopy Data

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Routine Validation of Gamma Spectroscopy Data

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List of Acronyms and Abbreviations

CLP	Contract Laboratory Program (EPA)
COC	chain of custody
DER	duplicate error ratio
DLC	decision level concentration
EPA	US Environmental Protection Agency
ER	environmental restoration
FSF	Field Support Facility
LANL	Los Alamos National Laboratory
LCS	laboratory control sample
n/a	not applicable
N.A.	not available
%R	percent recovery
QC	quality control
RER	replicate error ratio
RN	request number
σ	sigma (standard deviation of a set of measurements)
SMO	Sample Management Office
SOP	standard operating procedure
SOW	statement of work
TPU	total propagated uncertainty

Routine Validation of Gamma Spectroscopy Data

NOTE: Environmental Restoration (ER) Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/documents/Procedures/sops.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

1.0 PURPOSE

This standard operating procedure (SOP) represents the minimum standard for evaluating gamma spectroscopy data generated for the Los Alamos National Laboratory (LANL) ER Project for samples analyzed for gamma-emitting isotopes using the methods required under the current statement of work (SOW) for analytical services (LANL 1995). The evaluation of data by this procedure is not specific to a particular data use, although this procedure may be used as a point of departure for developing focused data validation requirements specific to a particular data use.

Note: Implementation of this procedure results in a tabulation of data compliances and noncompliances identified relative to expectations based on national guidelines (EPA 1994) for data review. Because the US Environmental Protection Agency (EPA) guidelines are specific to analyses for inorganic chemicals, additional guidance (ANSI 1996, Currie 1968, Fong and Alvarez 1996, MARSSIM 1997, and LANL 2000) was employed in the preparation of this SOP. Because the acceptance criteria used for this procedure are not based on site-specific acceptance criteria, the results of this validation procedure are intended to be used as *general indicators* of data quality and should not be construed as a definitive identification of data usability.

Note: Implementation of this procedure may be followed by a more focused and data use-specific evaluation of data, especially if implementation of this SOP indicates that technical deficiencies may exist in the data.

2.0 TRAINING

All data validators who implement this SOP shall possess a minimum of a Bachelors degree in chemistry and two years experience in generating analytical data in an environmental or radiochemistry analytical laboratory, or two years of data-validation experience that includes the production or validation of gamma spectroscopy data. New validators shall work under the direct supervision of an experienced ER Project validator. The work of new validators shall be reviewed and signed by an experienced ER Project validator until ten data packages for

each analytical suite have been satisfactorily validated. ER Project validators shall have demonstrated familiarity with the EPA national functional guidelines for data review. All data validators must document that they have read and understand this SOP and completed all applicable training assignments in accordance with QP-2.2.

3.0 DEFINITIONS

3.1 *Activity concentration* — Level of radioactivity per unit volume or mass measured as a concentration; usually reported in pCi/g or pCi/L.

3.2 *Analyte* — The element, nuclide, or ion a chemical analysis seeks to identify and/or quantify; the chemical constituent of interest.

3.3 *Annihilation radiation* — The two gamma rays of 0.511 MeV energy that are emitted as a result of the combination and disappearance of an electron and a positron.

3.4 *Anthropogenic* — Refers to environmental alterations that result from the presence or activities of humans.

3.5 *a posteriori* — In this SOP, defined as "after the measurement."

3.6 *a priori* — In this SOP, defined as "before the measurement."

3.7 *Blank sample* — Sample expected to have negligible or unmeasurable amounts of analytes. Results of blank-sample analyses indicate whether field samples might have been contaminated during the sample collection, transport, storage, preparation, and analysis process.

3.8 *Data validator* — A person who has met the minimum standards of training established by the ER Project for data validation and who performs data validation on behalf of the ER Project.

3.9 *Decision level concentration* — Activity concentration level used to classify radiochemical measurements as "detected" or "nondetected" (e.g., measured results above the DLC are "detected"). The DLC is established from an appropriate blank count at the 0.05-significance level. Therefore an observed activity concentration above the DLC has a less than 5% chance of originating from a sample containing no (or blank) levels of analyte.

$$DLC = C \times 2.33\sqrt{N_b} = C \times 2.33 S_b .$$

Where:

DLC = decision level concentration reported in pCi/g or pCi/L,

C = a group of factors that convert counts to an activity concentration (C is omitted if S_b is expressed in concentration units),

2.33 = 1.65 $(2)^{0.5}$ (1.65 normal probability, one sided, for 0.05 significance),

N_b = total analyte-free blank (or background) count,

S_b = standard deviation of the blank count, and
all blank, background, and sample count times are equal.

3.10 *Detect (radionuclides)* — Sample result greater than the MDC reported by the analytical laboratory. The laboratory reports the concentration of the analyte in the sample.

3.11 *Detector background* — Ambient signal response, recorded by radioactivity measuring instruments, that is independent of radioactivity contributed by the radionuclides being measured in the sample.

3.12 *Duplicate analysis* — Analysis performed on one of a pair of identically prepared subsamples taken from the same sample.

3.13 *Duplicate error ratio (DER)* — See replicate error ratio (RER).

3.14 *Holding time* — The maximum elapse of time that one can expect to store a sample without unacceptable changes in analyte concentrations. Holding times apply under prescribed conditions and deviations from these conditions may affect the holding time. Extraction holding time refers to the time lapse from sample collection to sample preparation; analytical holding time refers to the time lapse between sample preparation and analysis.

3.15 *Gamma spectroscopy data* — Analytical results and associated data for samples analyzed for gamma-emitting isotopes. Routine validation of gamma spectroscopy is included in this and not in ER-SOP-15.07 because of the greater complexity of gamma spectroscopy data.

3.16 *Laboratory control sample (LCS)* — A known matrix that has been spiked with compound(s) representative of the target analytes. The LCS is used to document laboratory performance. The acceptance criteria for LCSs are method specific.

3.17 *Laboratory duplicate sample* — The portions of a sample taken from the same sample container, prepared for analysis and analyzed independently but under identical conditions; used to assess or demonstrate acceptable laboratory method precision at the time of analysis. Each duplicate sample is expected to be equally representative of the original material. Duplicate analyses also are performed to generate data, to determine the long-term precision of an analytical method on various matrices.

3.18 *LANL data validation qualifiers* — The data qualifiers defined by LANL and used in the ER Project baseline-validation process. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate ER Project SOP.

3.19 *LANL data validation reason codes* — The codes applied to the sample data by data validators who are independent of the contract laboratory which performed the sample analysis. Reason codes provide an in-depth and

analysis-specific explanation for applying the qualifier with some description of the potential impact on the data use. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate ER Project SOP.

3.20 *Matrix spike* — An aliquot of sample spiked with a known concentration of target analyte(s). Matrix-spike samples are used to measure the ability to recover prescribed analytes from a native sample matrix. The spiking typically occurs before sample preparation and analysis.

3.21 *Minimum detectable concentration* — Minimum activity concentration that the analytical laboratory equipment can detect in 95% of the analyzed samples. That is, if the actual concentration of a sample is above MDC, a less than a 5% chance exists that the measured concentration will fall below the DLC and result in a "nondetect." An MDC measures analytical performance (not detection limits).

$$MDC = C \times (2.71 + 4.65\sqrt{N_b}) .$$

Where:

MDC = minimum detectable concentration reported in pCi/g or pCi/L,

C = a group of factors that convert counts to an activity concentration (C is omitted if S_b is expressed in concentration units),

2.71 = 1.65^2 (1.65 normal probability, one sided, for 0.05 significance),

4.65 = $1.65 \times 2(2)^{0.5;}$

N_b = total analyte-free blank (or background) counts, and

all blank, background, and sample count times are equal.

3.22 *Nondetect* – A sample result that is less than the MDC.

3.23 *Percent recovery (%R)* — Amount of material detected in a sample (minus any amount already in the sample) divided by the amount added to the sample and expressed as a percentage.

3.24 *Precision* — Concept used to describe dispersion of measurements. Precision may be absolute or relative to a particular measure of central tendency. The mathematical formulas used to determine precision vary according to the problem at hand.

3.25 *Preparation blank* — An analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in the environmental sample processing, and which is prepared and analyzed in the same manner as the corresponding environmental samples. The preparation blank is used to assess the potential for contamination of samples during preparation and analysis.

3.26 *Replicate error ratio* – Measure of precision of analytical laboratory replicate samples in a batch. The RER is based on the standard deviations of the sample and the replicate sample.

$$\text{RER} = \frac{S - R}{\sqrt{u_S^2 + u_R^2}}.$$

Where:

RER = replicate error ratio,
S = sample value,
R = replicate value,
u_S = sample uncertainty, and
u_R = replicate uncertainty.

If |RER| < 2, then the sample and replicate are not statistically different at the 95% confidence level.

3.27 *Request number (RN)* — An identifying number assigned by the ER Project to a group of samples that are submitted for analysis.

3.28 *Routine data* — Data generated using analytical methods that are identified as routine methods in the current ER Project SOW for analytical services.

3.29 *Routine data validation* — The process of reviewing analytical data relative to quantitative routine acceptance criteria. The objective of routine data validation is two-fold: one objective is to estimate the technical quality of the data relative to minimum national guidelines adopted by the ER Project; the other objective is to indicate to data users the technical data quality at a general level by assigning qualifier flags to environmental data whose quality indicators do not meet acceptance criteria.

3.30 *Sigma (s)* — Standard deviation (square root of the variance) of a set of measurements. For normally distributed data, a range of one sigma (1σ) below the estimated mean to one sigma (1σ) above the estimated mean signifies a 67% confidence that the mean of a population lies within that range. Similarly, a range of plus/minus 2 sigma (±2σ) implies 95% confidence that a population mean lies within that range.

3.31 *Target analyte* — An element, chemical, or parameter, the concentration, mass, or magnitude of which is designed to be quantified by use of a particular test method.

3.32 *Total propagated uncertainty (TPU)* — Sum of all aspects of uncertainty introduced throughout the sample analysis process, from sample collection to reporting of results. Many aspects of TPU may be specifically calculated by an analytical laboratory (e.g., net instrumental error, counting uncertainty). Other aspects of TPU may not be quantifiable (e.g., heterogeneity of

concentrations at site), and thus cannot be directly included in a laboratory's estimate of TPU.

4.0 BACKGROUND AND PRECAUTIONS

4.1 The ER Project analytical services SOW requires a suite of 43 nuclides in the gamma spectroscopy list (LANL 1995). The data-qualification system for this SOP divides the 43 nuclides into five groups which have common characteristics as related to measurement by gamma spectroscopy.

4.1.1 These five groups are defined in Table 4.1-1 (next page). The scope of this SOP is to provide a baseline quality review of the data in first group of analytes listed in Table 4.1-1. The first group are the nuclides of greatest potential concern for the ER Project.

4.1.2 The scope of this SOP includes a comparison of sample results with a statistical test for detection of each nuclide, a comparison of sample results with the blank result, and a review of quality control (QC) results for duplicate and LCSs. The comparison of the results with the decision level and the blank results is conducted for all 43 nuclides.

4.1.3 Additional tests in this SOP are only applied to the nuclides in Group 1 (those that are of greatest interest to the ER Project).

4.1.4 The scope of this SOP does not include a complete evaluation of all of the reported results because there are many factors to consider when reviewing gamma spectroscopy data that are beyond the scope of this SOP. These additional factors require the professional judgement and knowledge of an experienced radiochemist to interpret the usefulness of the reported data. Therefore, results qualified by this SOP as PM or RPM must be carefully and expertly reviewed by a qualified chemist before any use in order to accurately reflect the value of results from gamma spectroscopy analyses.

Table 4.1-1*
Grouping of ER Project Gamma Spectroscopy Suite Nuclides

Group #	Analytes		Data Quality Evaluation Approach
1	Am-241 Co-60 Cs-134 Cs-137	Eu-152 Na-22 Ru-106 U-235	Nuclides are evaluated as potential historical contaminants. Therefore, the highest level of data validation is required
2	Ce-144 Co-57 Mn-54	Pa-233 Se-75 Zn-65	Nuclides have anthropogenic origins, but have a half-life of less than 365 days and are not typically evaluated as primary nuclides. Data use is based on the professional judgment of an experienced radiochemist.
3	Ac-228 Ba-140 Bi-212 I-129 La-140 Np-237 Pa-231	Pa-234m Pb-210 Pb-211 Ra-223 Ra-224 Ra-226 Rn-219	Nuclides are not reliably measured by gamma spectroscopy. Data use must be based on the professional judgment of an experienced radiochemist. Manual review of the raw data is required before use as primary nuclides.
4	Bi-211 Bi-214 K-40 Pb-212 Pb-214	Th-227 Th-234 Tl-208 Annihilation radiation	Nuclides are naturally occurring. Data use must be based the on professional judgment of an experienced radiochemist.
5	Cd-109 Ce-139 Hg-203	Sn-113 Sr-85 Y-88	Radionuclide measured by gamma spectroscopy for quality-control purposes only. Data use must be based on the professional judgment of an experienced radiochemist.

* Source: Vanden Plas 2000.

4.2 To protect the integrity of the data record package, the **data validator** must store and handle all data record packages under ER Project chain-of-custody (COC) rules in accordance with ER-SOP-15.09.

4.3 Logic diagrams that appear in this SOP are included for experienced validators to expedite the validation process and do not include instructions for where to record validation results. Those instructions may be found in the SOP text that corresponds to each logic diagram.

4.4 The gamma spectroscopy data validation checklist forms require actions to be taken if a particular validation condition is true or false. It is important to look at the top of each validation form to know whether action is required when the condition is true or when the condition is false.

4.5 The validation process requires that the **data validator** record LANL data validation qualifiers and reason codes on photocopies of the data summary results forms ("Form Is") in the hard copy data record packages. Contiguous lines of identical qualification on the photocopied Form Is may be represented as the qualifier flag and reason code, followed by a vertical downward arrow to the end of the block of results that are qualified identically.

4.6 The gamma spectroscopy data validation checklist forms of Attachment D are examples of the actual forms to be used for data validation under this SOP. Individual parts of the forms may be reproduced as necessary to complete the validation of a data record package.

5.0 EQUIPMENT

The **validator** may need the following equipment and supplies to implement this procedure:

5.1 current gamma spectroscopy data validation checklist forms (see examples in Attachment D),

5.2 data record packages to be validated,

5.3 electronic calculator (optional),

5.4 photocopier, and

5.5 current ER Project SOW for analytical services.

6.0 PROCEDURE

Note: Deviations from this SOP are made in accordance with QP-4.2.

Note: While this SOP is applicable to all 43 of the nuclides in the gamma spectroscopy suite, the nuclides are treated differently by this procedure depending on the reliability and usefulness of the data produced by gamma spectroscopy for each nuclide. Included in Table 4.1-1 are the five different groups of nuclides that comprise the gamma spectroscopy suite.

6.1 Prepare for Data Validation

6.1.1 The **validator** will begin by obtaining the required current versions of the gamma spectroscopy data validation checklist forms (see Attachment D) from the ER Project website (http://erinternal.lanl.gov/Quality/forms.htm).

6.1.2 Obtain from the Sample Management Office (SMO) of the Field Support Facility (FSF) the data record package(s) that contain the sample data to be validated.

6.1.3 Prepare a data validation cover sheet (see Attachment C) by filling out the top part of the form and placing a check or other mark adjacent to the analytical suite(s) for which this validation is being performed.

Note: A single cover sheet may be used for validation of multiple analytical suites under the same RN.

Note: Use a separate sheet of paper to document each deficiency identified beyond the scope of this procedure including phone conversations with the analytical laboratory personnel concerning these deficiencies. Attach these sheets to the data validation cover sheet.

6.1.4 Verify that the following items are present in the data record package:

6.1.4.1 signed LANL COC record;

6.1.4.2 case narrative;

6.1.4.3 result forms (Contract Laboratory Program (CLP) Form I or equivalent) for each sample;

6.1.4.4 QC Forms (CLP form equivalents) for water and/or soils, as appropriate; and

6.1.4.5 the instrument readout (raw data) for the samples.

6.1.5 If the data record package does not contain all items listed in Sections 6.1.4.1 through 6.1.4.5, contact the analytical laboratory to obtain those materials.

6.1.5.1 If required documentation is missing from the data record package, and the package is less than six months old, contact the analytical laboratory and allow three business days for the laboratory to submit the required documentation.

6.1.5.2 If the analytical laboratory does not submit documentation within three business days, return the data record package to the SMO for contract-compliance action.

6.1.5.3 If the data record package is greater than 6 months old, allow 10 business days for the analytical laboratory to submit the required documentation before returning the data record package to the SMO.

6.1.6 Record the presence or absence (Y or N) of each item, as appropriate, in the completeness checklist of the data validation cover sheet.

6.1.7 In the data validation cover sheet completeness checklist section, note any samples whose data are missing from the data record package.

6.1.8 Photocopy all analytical laboratory QC forms from the data record package.

6.1.9 Photocopy the case narrative from the data record package.

6.1.10 Photocopy the Form Is to be used during the validation process before you begin completing this procedure.

Caution: Do not record data-validation qualifiers and reason codes on the original form (Form Is).

Note: The **validator** must submit photocopies of the items listed in Sections 6.1.8 through 6.1.10 as attachments to the completed data validation checklists.

6.2 Statistically Validate Sample Results and Verify Duplicate Error Ratio and Precision

Note: Section 6.2 applies to all gamma spectroscopy nuclides. The statistical analysis must be performed first in order to determine the detection status for the target nuclides before completing the subsequent steps in this validation procedure.

$$MDC = C \times (2.71 + 4.65\sqrt{N_b}) \qquad \text{(Equation 1)}$$

6.2.1 If a MDC *was stated* in the report for each nuclide in each batch associated with this RN,

6.2.1.1 record “Y” in block 1a of the gamma spectroscopy data validation checklist, Part Ia;

6.2.1.2 record the applicable nuclides and MDCs in block 1c of the gamma spectroscopy data validation checklist, Part Ia;

6.2.1.3 record “n/a” in blocks 2a, 2c, 3a, and 3c of the gamma spectroscopy data validation checklist, Part Ia, and

6.2.1.4 go to Section 6.2.5.

6.2.2 If a MDC *was not stated* in the report for each nuclide in each batch associated with this RN,

6.2.2.1 record “N” in block 1a of the gamma spectroscopy data validation checklist, Part Ia and

6.2.2.2 record “N.A.” in block 1c of the gamma spectroscopy data validation checklist Part Ia.

6.2.3 If the MDC *is not available* in the data record package,

6.2.3.1 record “Y” in block 2a of the gamma spectroscopy data validation checklist, Part Ia;

6.2.3.2 calculate an estimated sample-specific MDC as 3 × TPU of the sample result;

6.2.3.3 record this value appropriately in block 2c of the gamma spectroscopy data validation checklist, Part Ia;

6.2.4 If the sample value is greater than or equal to the MDC,

6.2.4.1 record a “Y” in block 3a of the gamma spectroscopy data validation checklist, Part Ia;

6.2.4.2 circle the appropriate qualifier in block 3b of the gamma spectroscopy data validation checklist, Part Ia;

6.2.4.3 record the appropriate qualifier flag and reason code combination next to the result for each affected target analyte, on Form I; and

6.2.4.4 record what analytes were qualified in block 3c of the gamma spectroscopy data validation checklist, Part Ia,

6.2.5 If the sample value is not greater than or equal to the MDC,

6.2.5.1 record “Y” in block 4a of the gamma spectroscopy data validation checklist Part Ia;

1) circle the qualifier flag and reason code combination “U, R5” in block 4b of the gamma spectroscopy data validation checklist, Part Ia;

2) record the qualifier flag and reason code combination “U, R5” next to the result for each affected target analyte, on Form I; and

3) record what analytes were qualified in block 4c of the gamma spectroscopy data validation checklist Part Ia.

Note: Sections 6.2.6–6.2.8 only apply to those gamma-spectroscopy analytes in Group 1 (see Table 4.2-1).

6.2.6 Find the reported DER in the data record package for the duplicate and the sample result, or calculate the DER (or RER) as follows:

$$\text{RER} = \frac{S - R}{\sqrt{u_S^2 + u_R^2}}. \qquad \text{(Equation 2)}$$

Where:

RER = replicate error ratio,

S = sample value,

R = replicate value,

u_S = sample uncertainty, and

u_R = replicate uncertainty.

If |RER| is less than two, then the sample and replicate are not statistically different at the 95% confidence level.

6.2.7 If the DER is less than two for the duplicate *and* the sample result,

6.2.7.1 record a “Y” in block 1a of the gamma spectroscopy data validation checklist, Part Ib and

6.2.7.2 go to Section 6.3, Verify Method Blank Results.

6.2.8 If the DER *is not* less than two for the duplicate *and* the sample result,

6.2.8.1 record “N” in block 1a of the gamma spectroscopy data validation checklist, Part Ib;

6.2.8.2 circle the qualifier flag and reason code combination “R, R7b” in block 1b of the gamma spectroscopy data validation checklist, Part Ib;

6.2.8.3 record the qualifier flag and reason code combination “R, R7b” next to the result for each affected target analyte, on Form I; and

6.2.8.4 record what analytes were qualified in block 1c of the gamma spectroscopy data validation checklist, Part Ib.

6.2.9 Use the logic diagram in Figure 6.2-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results based on the statistical analysis of the sample results and on the duplicate error ratio and the precision of the sample results.

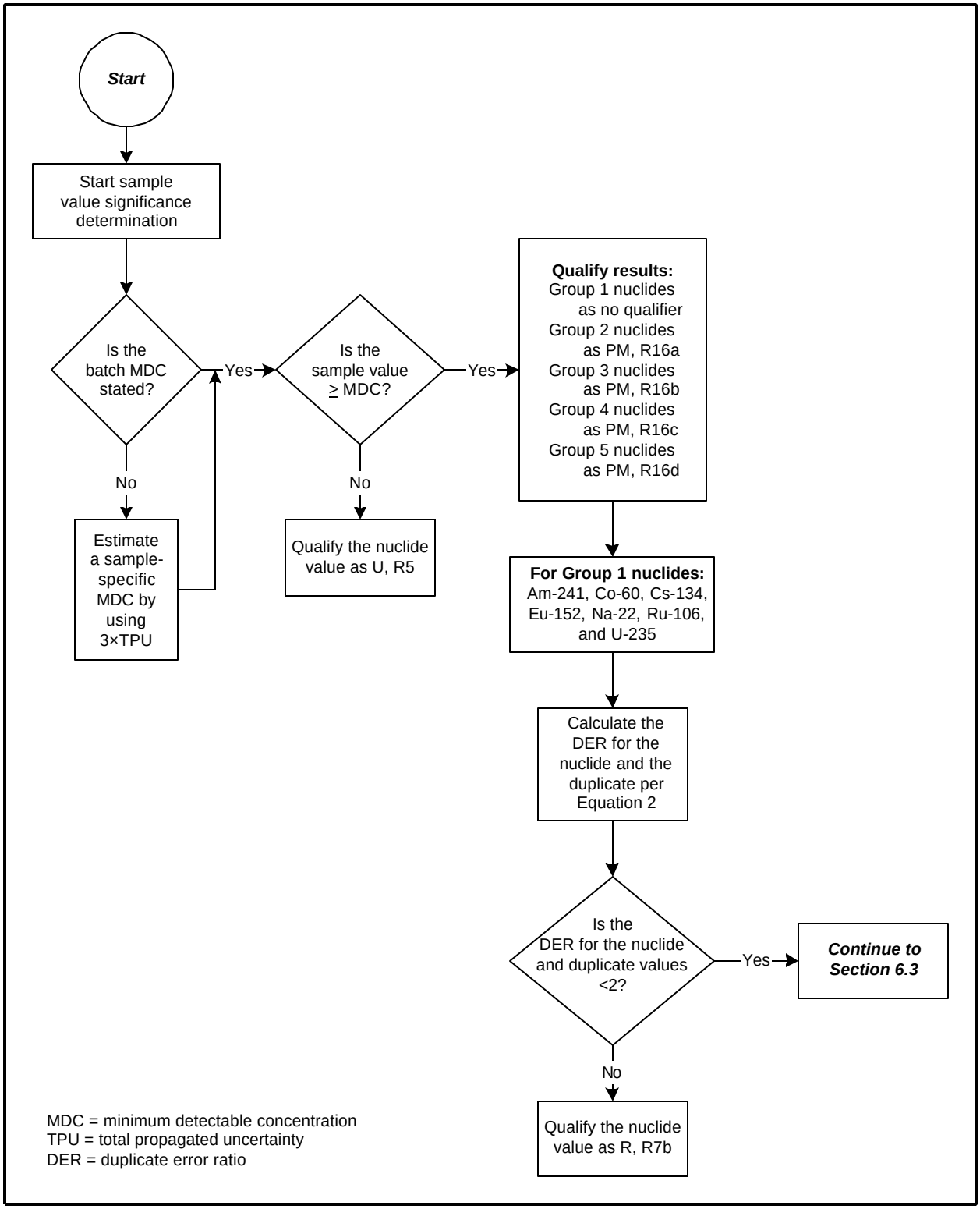

Figure 6.2-1. Assigning LANL qualifier flags and reason codes to the sample results based on the statistical analysis of the sample results and on the duplicate error ratio and the precision of the sample results.

6.3 Verify Method-Blank Results

Verify the presence of the required blanks and their associated results using forms provided by the analytical laboratory.

Note: Section 6.3 applies to all gamma spectroscopy analysis methods and analytes.

Note: If additional validation forms are needed to record validation data for more than one blank, make additional copies of the appropriate forms.

6.3.1 If a method blank *was analyzed* for each sample matrix *and* each specified nuclide in the batch,

6.3.1.1 record “Y” in block 1a of the gamma spectroscopy data validation checklist, Part IIa;

6.3.1.2 record “n/a” in block 1a of the gamma spectroscopy data validation checklist, Part IIa; and

6.3.1.3 go to Section 6.3.3.

6.3.2 If a method blank *was not analyzed* for each sample matrix and/or each specified nuclide in the batch,

6.3.2.1 record “N” in block 1a of the gamma spectroscopy data validation checklist, Part IIa;

6.3.2.2 circle “A, R4b” in block 1b of the gamma spectroscopy data validation checklist, Part IIa;

6.3.2.3 record the qualifier flag and reason code combination “A, R4b” next to the results of all samples for which a method blank was not analyzed, on Form I; and

6.3.2.4 record which sample matrix and/or specified nuclide(s) did not include a method-blank analysis in block 1c of the gamma spectroscopy data validation checklist, Part IIa. No further qualification is done.

6.3.3 For *each analyte*, if the method-blank result is less than the MDC or if the sample nuclide(s) result is greater than five times the blank result,

6.3.3.1 record “N” in block 2a of the gamma spectroscopy data validation checklist, Part IIb;

6.3.3.2 record “n/a” in blocks 2c and 2d of the gamma spectroscopy data validation checklist, Part IIb; and

6.3.3.3 go to Section 6.4 Verify Laboratory Control Sample Results.

6.3.4 If the sample nuclide(s) result is less than five times the blank result and the method-blank result is greater than the MDC (the sample result is undistinguishable from the blank),

6.3.4.1 record “Y” in block 2a of the gamma spectroscopy data validation checklist, Part IIb;

6.3.4.2 circle ”U, R4,” in block 2b of the gamma spectroscopy data validation checklist, Part IIb;

6.3.4.3 record the qualifier flag and reason code combination “U, R4” next to the result for each target analyte, on Form I;

6.3.4.4 record the detected blank analytes and affected samples in block 2c of the gamma spectroscopy data validation checklist, Part IIb; and

6.3.4.5 record the corresponding radionuclide activity in block 2d of the gamma spectroscopy data validation checklist, Part IIb.

6.3.5 Use the logic diagram of Figure 6.3-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant blanks.

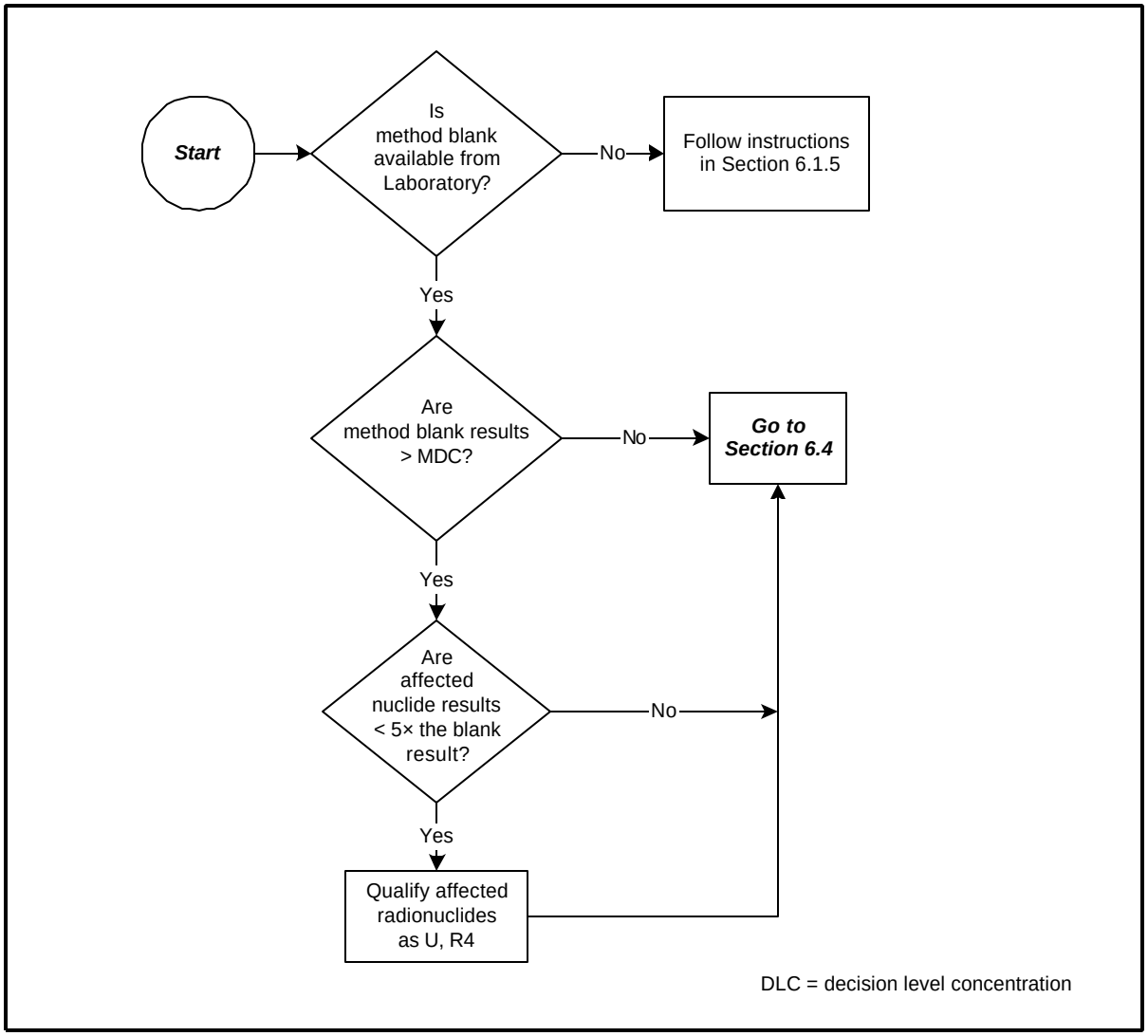

Figure 6.3-1. Assigning LANL qualifier flags and reason codes to the sample results for noncompliant blanks.

6.4 Verify Laboratory Control Sample Results

Note: Section 6.4 applies to all gamma spectroscopy analysis methods and those analytes included in the LCS. Verify the presence of the LCS sample %R values using forms provided by the analytical laboratory.

6.4.1 If an appropriate LCS *was analyzed* with this RN,

6.4.1.1 record “N” in block 1a, of the gamma spectroscopy data validation checklist, Part III;

6.4.1.2 record “n/a” in block 1c of the gamma spectroscopy data validation checklist, Part III; and

6.4.1.3 go to Section 6.4.3.

6.4.2 If an LCS *was not analyzed* with this RN,

6.4.2.1 record “Y” in block 1a, of the gamma spectroscopy data validation checklist, Part III;

6.4.2.2 circle "A, R6e" in block 1b of the gamma spectroscopy data validation checklist, Part III;

6.4.2.3 record the qualifier flag and reason code combination “A, R6e” next to the result for each affected target analyte, on Form I; and

6.4.2.4 record the LCS and/or LCS analytes not analyzed with this RN in block 1c of the gamma spectroscopy data validation checklist, Part III.

6.4.3 If the LCS %R values for *all analytes* fall between 80% and 120%, inclusive,

6.4.3.1 record “N” in blocks 2a and 3a of the gamma spectroscopy data validation checklist, Part III;

6.4.3.2 record “n/a” in blocks 2c, 2d, 3c and 3d of the gamma spectroscopy data validation checklist, Part III; and

6.4.3.3 go to Section 6.5, Assemble the Validation Data Record Package.

6.4.4 If *no* LCS analyte %R value is less than 80%,

6.4.4.1 record “N” in block 2a of the gamma spectroscopy data validation checklist, Part III;

6.4.4.2 record “n/a” in blocks 2c and 2d of the gamma spectroscopy data validation checklist, Part III; and

6.4.4.3 go to Section 6.4.6.

6.4.5 If *any* LCS analyte %R value is less than 80%,

6.4.5.1 record “Y” in block 2a of the gamma spectroscopy data validation checklist, Part III;

6.4.5.2 circle, in block 2b of the gamma spectroscopy data validation checklist, Part III,

1) “J-, R6a” for detected sample analytes and

2) “UJ, R6b” for nondetected analytes;

6.4.5.3 record, next to the result for each affected target analyte, on Form I, the qualifier flag and reason code combination

1) “J- ,R6a” for detected and

2) “UJ, R6b” for nondetected analytes;

6.4.5.4 record the noncompliant LCS analytes in block 2c of gamma spectroscopy data validation checklist, Part III; and

6.4.5.5 record the %R values of the noncompliant LCS analytes in block 2d of gamma spectroscopy data validation checklist Part III.

6.4.6 If *no* LCS analyte %R value is greater than 120%,

6.4.6.1 record “N” in block 3a of the gamma spectroscopy data validation checklist, Part V;

6.4.6.2 record “n/a” in blocks 3c and 3d of the gamma spectroscopy data validation checklist, Part V; and

6.4.6.3 go to Section 6.5, Assemble the Validation Data Record Package.

6.4.7 If *any* LCS analyte %R value is greater than 120%,

6.4.7.1 record “Y” in block 3a of the gamma spectroscopy data validation checklist, Part III;

6.4.7.2 circle, in block 3b of the gamma spectroscopy data validation checklist, Part III,

1) “J+,R6” for *detected* sample analytes and

2) no qualification for *nondetected* analytes;

6.4.7.3 for detected analytes, record the qualifier flag and reason code combination "J+, R6” next to the result for each affected target analyte, on Form I;

6.4.7.4 record the noncompliant LCS analytes in block 3c of gamma spectroscopy data validation checklist, Part III; and

6.4.7.5 record the %R values of the noncompliant LCS analytes in block 3d of gamma spectroscopy data validation checklist, Part III.

6.4.8 Use the logic diagram of Figure 6.4-1 determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant LCS analytes.

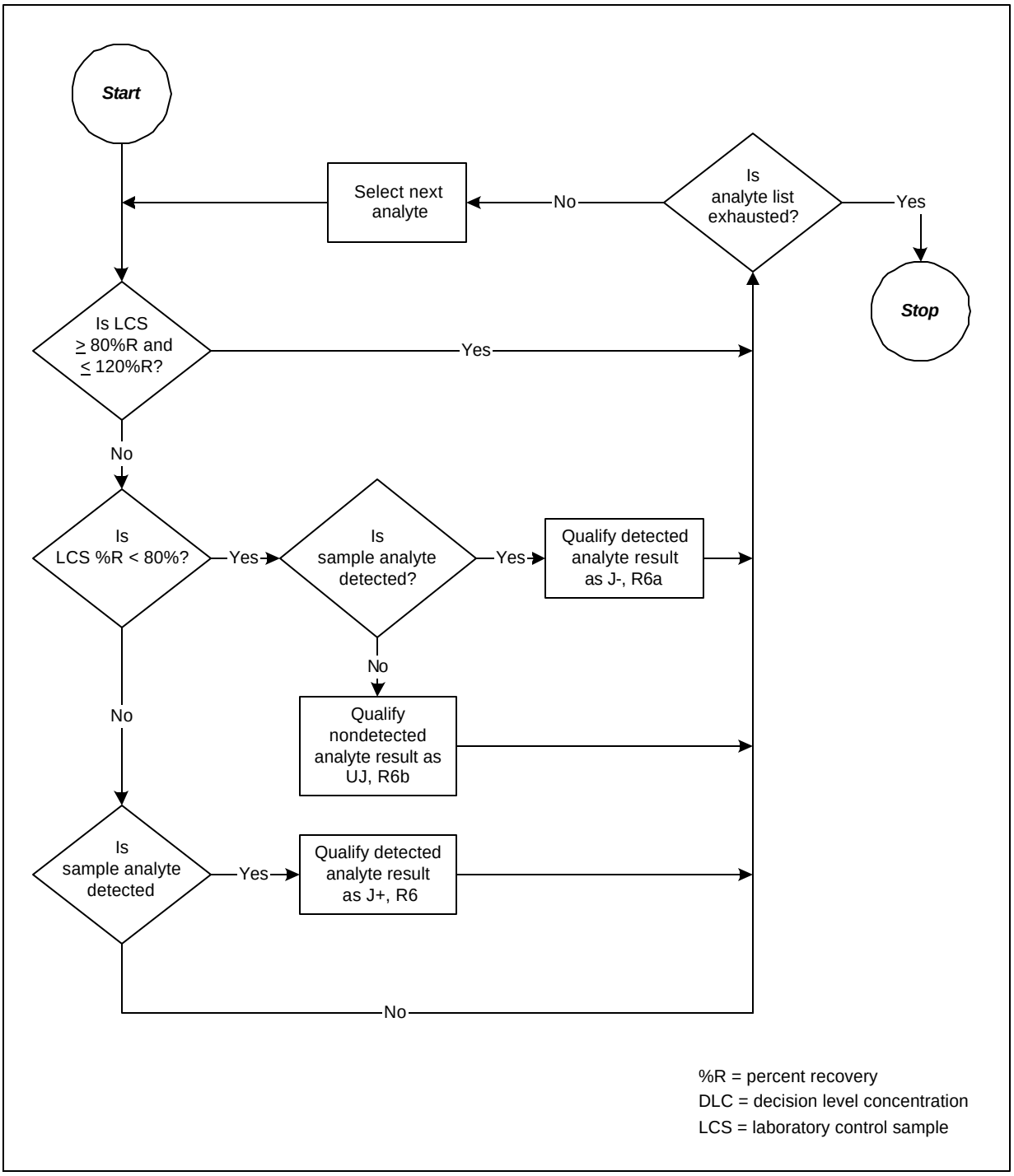

Figure 6.4-1. Assigning LANL qualifier flags and reason codes to the sample results for noncompliant LCS analytes.

6.5 Assemble the validation data record package to include the following items in the order they are listed below:

6.5.1 the completed, signed, and dated data validation cover sheet;

6.5.2 the gamma spectroscopy data validation checklists completed in Sections 6.2 through 6.4;

6.5.3 photocopies of the completed forms (Form Is) on which the data validator recorded the qualifier flags and reason codes;

6.5.4 a photocopy of the data record package case narrative; and

6.5.5 photocopies of the data record package QC forms (assemble in order by QC forms).

6.6 Submit the validation data record package to the SMO, in accordance with ER-SOP-15.09.

7.0 REFERENCES

The following documents have been cited within this procedure:

ANSI 1996. Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories, ANSI N42.23-1996.

Currie, L. 1968. “Limits for Qualitative Detection and Quantitative Determination”. *Analytical Chemistry*, (Vol. 40, No.3, pp. 586-593.) March, 1968

DOE (U.S. Department of Energy), December 1997. Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM), Final, Washington, D.C. (DOE 1997, 63128)

EPA (US Environmental Protection Agency), February 1994. “US EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review,” Publication 9240.1-05-01, EPA-540/R-94/013, Office of Solid Waste and Emergency Response, Washington, DC.

ER-SOP-15.09, Chain of Custody for Analytical Data Packages

Fong, S., and Alvarez, J. 1997. “Data Quality Objectives for Surface-Soil Cleanup Operation Using *In Situ* Gamma Spectrometry for Concentration Measurements.” *Health Physics*, (Vol. 72, No.2) February 1997.

LANL (Los Alamos National Laboratory), July 1995. “Environmental Restoration Project Statement of Work for Analytical Services,” Revision 2, RFP Number 9-SX1-Q4257, Los Alamos National Laboratory, Los Alamos, New Mexico.

QP-2.2, Personnel Orientation and Training

QP-4.2, Standard Operating Procedure Development

Vanden Plas, B., 2000. “Approach to Gamma Spectroscopy Data Quality Evaluation,” LA-UR-00-1088, Los Alamos National Laboratory, Los Alamos, New Mexico (ER2000-0061).

8.0 RECORDS

Although no records will be submitted to the Records Processing Facility (RPF) in the course of completing this procedure, the items identified in Section 6.5 will be a part of the data record package submitted to the RPF from the SMO in accordance with ER-SOP-15.09.

9.0 ATTACHMENTS

The document user may employ documentation formats different from those attached to/named in this procedure—as long as the substituted formats in use provide, as a minimum, the information required in the official forms developed by the procedure.

Attachment A: Laboratory Data Validation Qualifier Flags (1 page)

Attachment B: Gamma Spectroscopy Data Validation Reason Codes (1 page)

Attachment C: Data Validation Cover Sheet (1 page)

Attachment D: Gamma Spectroscopy Data Validation Checklist, Part I–Part VI. (4 pages)

Laboratory Data Validation Qualifier Flags

A	The contractually required supporting documentation for this datum is absent.
U	The analyte is classified as “not detected.”
J	The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual.
J+	The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual with a potential positive bias.
J-	The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual with a potential negative bias.
UJ	The analyte is classified as “not detected” with an expectation that the reported result is more uncertain than usual.
RPM	The reported sample result is classified as “rejected” due to serious non-compliances regarding quality control acceptance criteria. The presence or absence of the analyte cannot be verified based on routine validation alone.
PM	Manual review of raw data is recommended to determine if the observed non-compliance(s) with quality acceptance criteria adversely impacts data use.

Note: A “PM” qualifier flag indicates that a manual review should be conducted if the datum that is qualified with the “PM” is important to the data user. In addition, "PM" also means that a decision must be made by the project manager/delegee regarding the need for further review of the data. This review should include some consideration of potential impact that could result from using the "PM" qualified data.

Gamma Spectroscopy Data Validation Reason Codes

R4 The sample result is greater than the MDC but is less than five times the amount found in the blank.

R4b Blank data is either missing from or not reported in the data record package.

R5 Analyte is not detected because the amount reported is less than the MDC.

R6 Recovery of analyte in the LCS is greater than upper limit and the analyte is greater than the MDC in the sample.

R6a Recovery of analyte in the LCS is less than the lower limit and the analyte is greater than the MDC in the sample.

R6e The LCS data is missing from the data record package.

R7b The duplicate and sample results have a DER (duplicate error ratio) that is greater than 2.0.

R16a Result is greater than the MDC for the following fission and activation products with half-lives less than 365 days: Ce-144, Co-57, Mn-54, Pa-233, Se-75, and Zn-65.

R16b Result is greater than the MDC for the following radionuclides not reliably measured by gamma spectroscopy: Ac-228, Ba-140, Bi-212, I-129, La-140, Np-237, Pa-231, Pa-234, Pb-210, Pb-211, Ra,-223, Ra-224, Ra-226, and Rn-219.

R16c Result is greater than the MDC for the following naturally occurring radionuclides that are reliably measured by gamma spectroscopy and that can provide an indication of the quality of the gamma spectroscopy measurement: Bi-211, Bi-214, K-40, Pb-212, Pb-214, Th-227, Th-234, Tl-208, and annihilation radiation.

R16d Result is greater than the MDC for the following six radionuclides typically used by the analytical labs in their LCSs for instrument calibration and checks on instrument performance: Cd-109, Ce-139, Hg-203, Sn-113, Sr-85, and Y-88.

Data Validation Cover Sheet

Section I.

Request Number:_____ Validation Date:_____ Lab Code:_____

Contract Laboratory Name:_____

Validator:_____ Organization:_____

Analytical Suite (check all that apply): ☐ Volatile Organics ☐ Semivolatile Organics ☐ Organochlorine Pesticides/Polychlorinated Biphenyls ☐ High Explosives ☐ Inorganics ☐ Radiochemistry

Other (describe):_____

Section II. Completeness Check

Yes	No	n/a	(check one)	Yes	No	n/a	(check one)
☐	☐	☐	1. Chain-of-custody form(s)	☐	☐	☐	6. Raw/BSS data
☐	☐	☐	2. Case narrative	☐	☐	☐	7. Quality control forms
☐	☐	☐	3. Sample result forms	☐	☐	☐	8. Quantitation reports
☐	☐	☐	4. Sample chromatograms	☐	☐	☐	9. TICs forms
☐	☐	☐	5. Standard chromatograms	☐	☐	☐	10. TICs mass spectra

Identify any samples in the assigned Request Number that are missing:

_____ _____ _____ _____ _____ _____ _____ _____

_____ _____ _____ _____ _____ _____ _____ _____

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed upon date of resolution and contract laboratory point of contact):

(Attach additional comment sheets as necessary)

Validator's signature: _____ Date: _____

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Gamma Spectroscopy Data Validation Checklist

Part Ia. Sample Statistics Check

Criterion	Criterion true? (yes, no, or n/a)	Action if "criterion true?" = yes	Comment
Is the batch's MDC stated?	1a.	1b. Record the MDC in block 1c and go on to Section 6.2.3.	1c. MDC =
Is the batch's MDC *not* reported?	2a.	2b. Estimate the sample-specific MDC using the metric: MDC = 3 × TPU. Record the estimated MDC in block 2c.	2c. Estimated MDC =
Is the sample value = the MDC?	3a.	3b. Assign the LANL qualifier and reason code combinations as follows: Group 1 nuclides: no qualifier Group 2 nuclides: **PM, R16a** Group 3 nuclides: **PM, R16b** Group 4 nuclides: **PM, R16c** Group 5 nuclides: **PM, R16d** Record all qualified analytes in block 3c.	3c.
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Gamma Spectroscopy Data Validation Checklist

Part Ia. Sample Statistics Check (continued)

Criterion	Criterion true? (yes or no)	Action if "criterion true?" = yes	Comment
Is the sample value < the MDC?	4a.	4b. Assign the LANL qualifier and reason code combination "**U, R5**" to the affected nuclides. Record those analytes qualified "U" in block 4c.	4c.

Part Ib. Sample Statistics Check (continued)

Criterion	Criterion true? (yes or no)	Action if "criterion true?" = no	Comment
For Group 1 nuclides (Am-241, Co-60, Cs-134, Eu-152, Na-22, Ru-106, and U-235), is the DER for the nuclide and the duplicate values <2?	1a.	1b. Assign the LANL qualifier and reason code combination "**R, R7b**" to the nuclide value. Record those nuclides qualified "R" in block 1c.	1c.

Gamma Spectroscopy Data Validation Checklist

Part IIa. Method Blank Validation Criteria

Criterion	Criterion true? (yes or no)	Action if "criterion true?" = no Assign qualifier & reason code…	List affected matricies or batches.
Was a method blank analyzed for each sample matrix and batch?	1a.	1b. "**A, R4b**" for any missing documentation. In block 1c, record all sample matrices and/or analytical batches that did not include a method blank.	1c.

Part IIb. Method Blank Validation Criteria (continued)

Criteria	Criterion true? (yes or no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List detected blank analyte(s) and affected samples.	Analyte concentration (pCi/g)
Is a target analyte detected in the method blank **AND** is the same target analyte detected in the sample result < five times the amount detected in the method blank?	2a.	2b. "**U, R4**" to affected sample analyte(s).	2c.	2d.

Gamma Spectroscopy Data Validation Checklist

Part III. Laboratory Control Sample (LCS) Validation Criteria

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List all noncompliant LCS analytes.	LCS %Rs
Was a required LCS *not* associated with this request?	1a.	1b. "**A, R6e**" for any missing documentation. In block 1c, record • any LCS analytes not reported.	1c.	1d. n/a
Is the LCS percent recovery value < 80% for any analyte?	2a.	2b. "**J-, R6a**" to all *detected* sample analytes and "**UJ, R6b**" to all *nondetected* sample analytes. In block 2c record • noncompliant LCS analytes. In block 2d record • the %R values of the noncompliant LCS analytes.	2c.	2d.
Is the LCS percent recovery value > 120% for any analyte?	3a.	3b. "**J+, R6**" to all *detected* sample analytes and do not qualify *nondetected* sample analytes. In block 3c record • noncompliant LCS analytes. In block 3d record • the %R values of the noncompliant LCS analytes.	3c.	3d.